S-5

Fabrication of $Nd_2Fe_{14}B$ Powders by Spray-Drying and Reduction–Diffusion Processes

Korea Institute of Machinery and Materials, C.J. Choi* and B.K. Kim
Shenyang Polytechnic University, P.R. China, X.L. Dong

1. Introduction

An intense effort has been made on research and development of Nd-Fe-B magnets (1). The reduction-diffusion (R/D) technique (2) was applied to prepare Nd-Fe-B alloy (3). It has been proved that the R/D process has many advantages in terms of energy consumption and production cost. Recently, we developed a new route by firstly adding a spray-drying process into the R/D fabrication of $Nd_2Fe_{14}B$-type alloy with the particle size below 1 μm (4). In this work, the effects of the processes on disintegration, phases, morphologies, microstructures and characteristics of the $Nd_2Fe_{14}B$-type powders were investigated.

2. Experimental Details

Spray-drying is the process of spraying a solution into a warm drying medium to produce nearly spherical powder granules that are relatively homogeneous (5). The starting raw materials of metal salts were weighted and dissolved into de-ionized water for the target stoichiometric composition of $Nd_2Fe_{14}B$. This solution was spray-dried to prepare the precursor, which was treated by a series of steps, i.e. debinding, milling, reducing and washing. The phases, morphologies, microstructures, compositions and thermal properties of the powders were identified by means of XRD, SEM, and TG-DTA.

3. Results and Discussion

XRD (Fig. 1) were performed on the powders obtained from the first step (i.e., spray-drying) to the final one (i.e., washing). It is shown that spray-dried precursor [Fig. 1(a)] is amorphous, whose morphologies have the spherical shell shape of 5-40 microns in diameter, with smooth or dimpled surfaces as shown in SEM micrograph of Fig. 2(a). The amorphous precursor was crystallized into finer oxides of Nd and Fe [Figs. 1(b) and 2(b)] by debinding during which the volatile components were removed. The resultant mixture is of chemically mixed powders. Milling treatment was carried out to triturate the aggregation of the powders. The milled powders were reduced under a hydrogen atmosphere, during which the crystallites of α-Fe were separated out from the aggregated Fe_2O_3 [Figs. 1(c) and 2(c)].

The H_2-reduced powders were mixed with granular calcium and compressed into a green compact with a relatively low pressure. The compact was used for Ca-reduction and diffusion,

which was proceeded under a flowing Ar atmosphere at 1000 °C for 2 hours. All constituents were reduced and transformed into alloy particles by the solid-state diffusion. The consequent product was a mixture of CaO and $Nd_2Fe_{14}B$ alloy [Fig. 1(d)], which was in a loosely interspersed with fine particles of the by-product CaO. A necessary subsequent process serves to chemically remove the excess calcium metal and the CaO. The production of this route immediately results in the formation of a single phase of $Nd_2Fe_{14}B$ compound [Fig. 1(e)]. For the purpose of clean removal of the residual CaO which would deteriorate magnetic properties of the powders, the washing treatment with dilute acetic acid and water was repeatedly used. The recovered $Nd_2Fe_{14}B$ particles are in sphere-shape with mean particle size less than 1 micron [Fig. 2(d)].

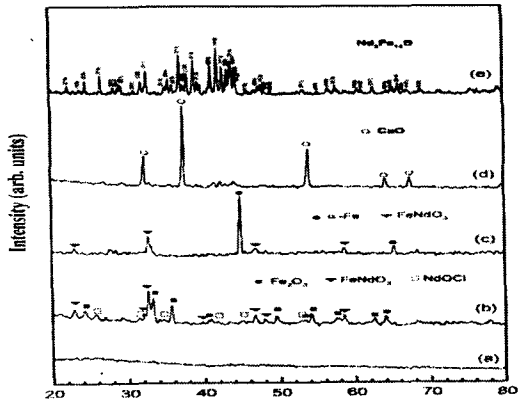

Figure 1. XRD patterns of the products in all step of the process: (a) spray-drying; (b) de-binding; (c hydrogen reducing; (d) calcium reducing and (e washing.

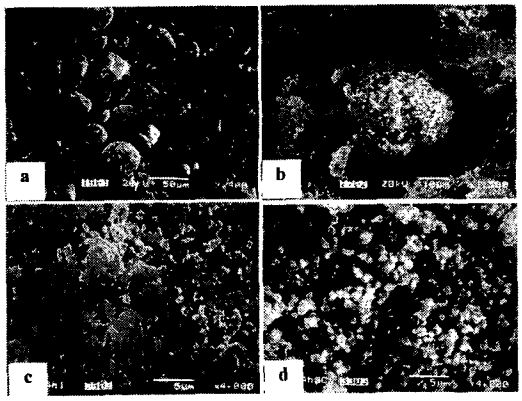

Figure 2. SEM micrographs of the powder (a) spray-drying; (b) de-binding; (c) milling an subsequently H_2-reducing; (d) Ca-reducing an subsequently washing.

4. Summary

The magnetic alloy of $Nd_2Fe_{14}B$-type was synthesized by a mechano-chemical method, including the processes of spray-drying, de-binding, milling, H_2- and Ca-reduction and washing. The very fine $Nd_2Fe_{14}B$ particles smaller than 1 μm could be achieved without the inevitable pulverization step in conventional processes.

References

[1] M. Sagawa, et al. J. Appl. Phys. 55, 2083 (1984).
[2] R.E. Cech, U.S. Patent, 3,625,779 (1971); J. Metals, **26**, 32 (1974).
[3] Y. Okajima, T. Tsugita, T. Takechi and S. Okada, U.S. Patent no 4,681,623 (1987).
[4] B.K. Kim, C.J. Choi and X.L. Dong, US patent, in review.
[5] S. Katta and W.H. Gauvin, J. AIChE, **21**, 1 (1975).